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INFLUENCES OF CO2 CONTINUOUS LASER PREPROCESSED SUBSTRATES ON OPTICAL THIN FILM DAMAGE THRESHOLDS[1]

Wu Zhouling Gao Yang[2] Fan Zhengxiu Wang Zhijiang

ABSTRACT

As far as experimental research into the influences of CO_2 laser preprocessed substrates on optical thin film damage threshold values is concerned, it was discovered that, with regard to single layer films and antireflective coatings, radiation pretreatment causes threshold values to clearly go up. At their highest, they reach 5 times those not radiation treated. However, on high reflection films, radiation pretreatment has no great influence. Using repetition frequency pulse photothermal deflection techniques, real time studies were done of corresponding relationships associated with light absorption and damage. Using pulsed time resolved light deflection technology, precise determinations were made of the locations associated with the occurrence of initial damage on samples in depth and direction. Making use of continuous modulation light deflection techniques--combining Nomarski optical microscope analysis of damage morphology--a number of significant conclusions were reached.

KEY TERMS: -- Thin film damage, Light absorption, Photothermal deflection, CO_2 laser pretreated substrate.

1. INTRODUCTION

Substrate surface quality has important influences on thin film optical characteristics. In order to lower thin film optical losses and raise the damage threshold values, we put a

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* Numbers in margins indicate foreign pagination. Commas in numbers indicate decimals.

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good deal of effort into substrate preparation and its pretreatment technology. This includes developing new polishing techniques [1-6], improving cleaning technology [7-10], drying substrates in a vacuum before plating films [11,12], as well as carrying out laser irradiation pretreatment on substrates [13-17], and so on. Although these efforts improved the quality of thin films at different levels, up to the present time, however, we still have not thoroughly gotten a clear understanding of the corresponding patterns between substrate preparation techniques and coating layer damage threshold values as well as their mechanisms.

This article uses TiO_2 and SiO_2 single layer films, antireflective coatings, as well as high reflection films plated on quartz substrates as examples and does experimental research on the influences of CO_2 laser pretreated substrates with regard to optical thin film absorption losses and damage threshold values. In conjunction with this, combining damage form analysis and photothermal deflection method real time detection of damage processes [18], analysis and discussion was done on the relevant mechanisms, and a number of significant conclusions were reached.

2. EXPERIMENTAL METHODS

Samples were uniformly plated onto quartz glass substrate. Actual film system structures and technical conditions were as shown in Fig.1. The CO_2 laser operating wave length used in order to carry out irradiation treatments was 10.6 microns. The power was approximately 120W. Irradiation facula diameter was 15mm. Irradiation time was 180s. With regard to carrying out irradiation treatment of substrates in air, after treatment, there was a 24h exposure to the air. Following this, they were put back in a vacuum and thin films were deposited. /330

However, in the case of carrying out irradiation of substrates in a vacuum, after irradiation, do not come in contact with air. They cool in a vacuum chamber, and, following that, there is direct deposition of film layers.

We carried out threshold value test measurements on experimental equipment [18] which we set up ourselves. Laser systems were composed of Nd:YAG oscillators and two stage Nd:YAG amplifiers. As far as oscillators are concerned, option was made for the use of LiF crystal Q modulation, small aperture diaphragm mode selection, output wave length being 1.06 micron, pulse width (FWHM) being 10ns, and operation in single mode status. Incident laser beams, through an anti-image error non spherical lens ($f \simeq 80mm$), converge onto sample surfaces. Facula diameter (I_0/e^2) is 44 microns. Damage tests opt for the use of 1-on-1 methods, that is, at the same location on sample surfaces, there is only one iteration of laser light--regardless of whether or not this point gives rise to damage. Thin film damage threshold values are defined as being initial damage threshold values corresponding to zero damage probability [19,20]. Compared to the traditional definition of threshold value as corresponding to 50% damage probability, this definition is capable of eliminating the influence of facula effects [21,22] during damage experiments and possesses obvious superiority. Thin film damage is defined as thin films going through laser irradiation after which they give rise to observable and irreversible physical changes. In this article, use is made of continuous modulation light deflection technology to observe damage [18,23]. The meaning of damage is thin film modulation light deflection signals producing irreversible transformations.

TABLE 1 INVESTIGATED SAMPLES AND PROCESSING TECHNOLOGIES (λ_0 = 1.06 micron).

1 样品编号	2 膜系结构	3 基板预处理工艺	4 膜层沉积工艺
S_{1a} S_{1b} S_{1c}	5 TiO_2 单层膜; $nd=\lambda_0/2$	6 常规处理 7 空气中激光辐照预处理 8 真空中激光辐照预处理	电子束热蒸发; 9 真空度 $(2\sim3)\times10^{-5}\times133.32$ Pa; 基板烘烤 200°C.
S_{2a} S_{2b} S_{2c}	5 SiO_2 单层膜; $nd=\lambda_0/2$	6 常规处理 7 空气中激光辐照预处理 8 真空中激光辐照预处理	
S_{3a} S_{3b} S_{3c}	A(LH)G H: TiO_2 L: SiO_2	6 常规处理 7 空气中激光辐照预处理 8 真空中激光辐照预处理	
S_{4a} S_{4b} S_{4c}	$A(HL)^{10}HG$ H: TiO_2 L: SiO_2	6 常规处理 7 空气中激光辐照预处理 8 真空中激光辐照预处理	

Key: (1) Sample Serial No. (2) Film System Structure (3) Substrate Pretreatment Techniques (4) Film Layer Deposition Techniques (5) Single Layer Film (6) Conventional Treatment (7) Laser Irradiation Pretreatment in Air (8) Laser Irradiation Pretreatment in a Vacuum (9) Electron Beam Thermal Evaporation; Degree of Vacuum (2-3) x 10^{-5} x 133.32 Pa; Substrate Drying 200°C.

Absorption measurements opt for the use of repetition frequency pulse light deflection techniques in order to carry out research. Going through analyses of the locations on the time axis of pulse light deflection signal peak values associated with critical destruction times, it is possible to precisely determine the locations of film layer surface, film layer interior, as well as film and substrate boundary layer damage [18].

As far as damage morphology is concerned, use is made of continuous modulation light deflection technology and Nomarski

optical microscopes to carry out comparative analyses. The former is capable of giving a concept of amount to damage degrees. The latter possesses the special point of unusual direct observation. The two combined are then an aid to analyzing damage behavior and relevant mechanisms associated with different film layers.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Sample Absorption and Damage Threshold Measurements

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Test measurement results were as shown in Table 2.

TABLE 2 MEASURED ABSORPTION AND LASER INDUCED DAMAGE THRESHOLDS OF THE SAMPLES INVESTIGATED (λ_0 = 1.06 micron).

1 项目 \ 2 结果	3 样			品		
	S_{1a}	S_{1b}	S_{1c}	S_{2a}	S_{2b}	S_{2c}
4 吸收率 $A(10^{-4})$	1.25±1.0	12.7±1.2	3.9±0.8	2.3±0.2	2.1±0.2	0.8±0.1
5 阈值 $F_{th}(Jcm^{-2})$	7.8±1.2	7.8±1.8	41.3±5.4	18.3±1.0	19.2±1.5	67.3±6.2
	S_{3a}	S_{3b}	S_{3c}	S_{4a}	S_{4b}	S_{4c}
4 吸收率 $A(10^{-4})$	11.3±0.9	10.4±1.1	4.2±1.8	6.8±1.3	6.6±1.8	7.1±1.5
5 阈值 $F_{th}(Jcm^{-2})$	4.3±1.8	4.4±1.3	18.2±5.7	8.8±2.1	8.2±3.1	8.6±2.5

Key: (1) Item (2) Result (3) Sample (4) Absorption Rate (5) Threshold Value

From Table 2, it is possible to see that:

(1) With regard to single layer films and antireflective coatings, in a vacuum, CO_2 laser pretreated substrates obviously raise damage threshold values. At the highest, they reach 5 times those of ones not irradiation treated. However, in the case of high reflection films, irradiation pretreated substrates, by contrast, have no great influence.

(2) In air, laser pretreated substrates do not raise film layer damage thresholds.

(3) For the same type of film system structure, damage threshold values and absorption possess clearly corresponding relationships. Absorptions are large and threshold values are small.

Our explanation of the phenomena above is that, with regard to TiO_2 and SiO_2 single layer films as well as antireflective films, the film layer-substrate boundary surface light fields are relatively strong. Absorption is comparatively large [16]. It is relatively easy to give rise to damage. CO_2 laser irradiation pretreatment in vacuums is capable of eliminating water vapor and other impurities adsorbed to substrate surfaces, improving film layer-substrate boundary surface quality (for example, lowering boundary surface absorption and so on). Thus, it is possible to raise single layer film and antireflective film damage threshold values. With regard to high reflection films, due to film layer-substrate boundary surface light fields being almost zero, the result is that, in the case of substrate irradiation treatment, it generally will not raise the damage threshold values. Substrate laser pretreatment in air does not raise film layer damage threshold values. The reason for this may lie in substrates--after irradiation treatment--producing

reoccurrences of adsorption of such impurities as water vapor and so on due to their re-exposure to the air.

3.2 Analysis of Sample Uniformity

Due to the influences of CO_2 laser beam light strength distributions when substrates are pretreated, relevant sample damage threshold values show clear nonuniformities within the entire region influenced by irradiation, as shown in Fig.1.

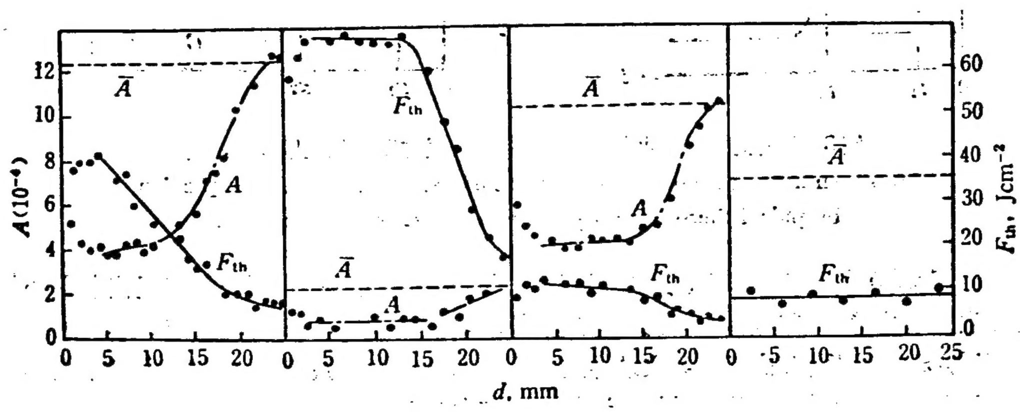


Fig.1 Uniformity of the samples investigated

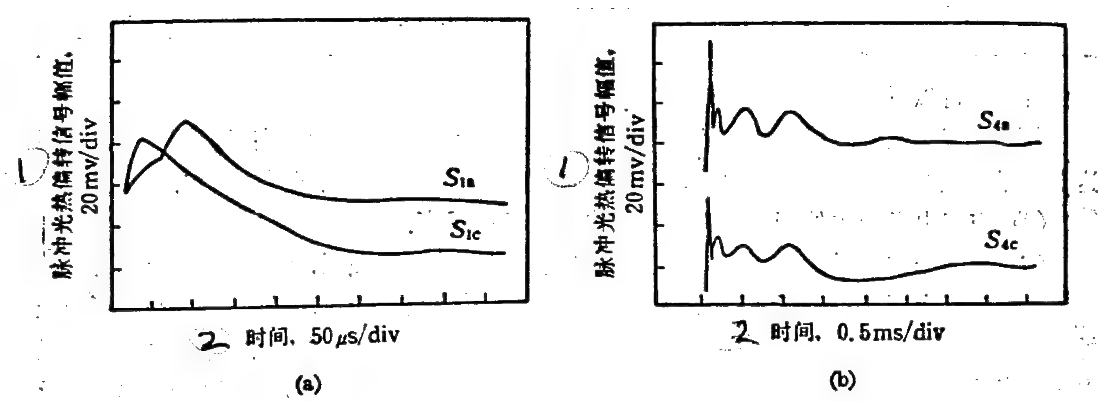
(1) Within a very small region close to the center of CO_2 laser irradiation, sample absorption is comparatively great, and threshold values are relatively low. The reason for this is that the substrate surface regions in question--during pretreatment processes--have already experienced CO_2 laser ablative destruction. /332

(2) After leaving the heavy absorption region associated with the center of irradiation, absorption follows increases in the distance from measurement points to the center of irradiation, first decreasing, and, afterward, increasing until reaching stability. However, threshold values, by contrast,

first increase and, afterward, decrease, finally also reaching stability. Between threshold values and damage, once again, good corresponding relationships were displayed. The explanation for this is that--during TiO_2 and SiO_2 film layer damage processes--absorption took the dominant role. This conclusion is in line with our earlier experimental results [12].

3.3 Sample Damage Process Studies

Fig.2 is time-resolved photothermal deflection signals at times of critical destruction associated with TiO_2 single layer films and TiO_2/SiO_2 high reflection films. Film layer light deflection signals produce sudden changes at times of critical destruction, causing the locations of peak signal values among time-resolved photothermal deflection signals to correspond to the locations where damage is produced in film layers [18] as shown in Fig.3.



(a) and TiO_2/SiO_2 HR Coating (b).

Fig.2 Pulsed time-resolved photothermal deflection signal of TiO_2 single layer

Key: (1) Pulse Photothermal Deflection Signal Peak Values (2) Time

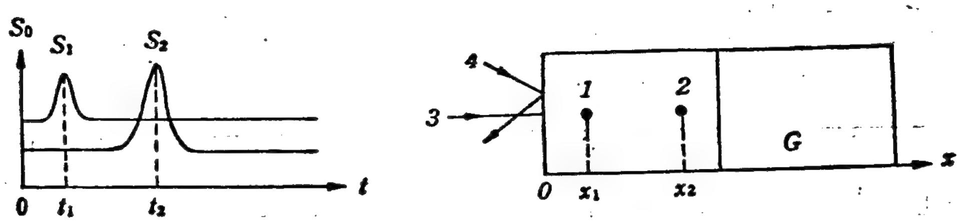
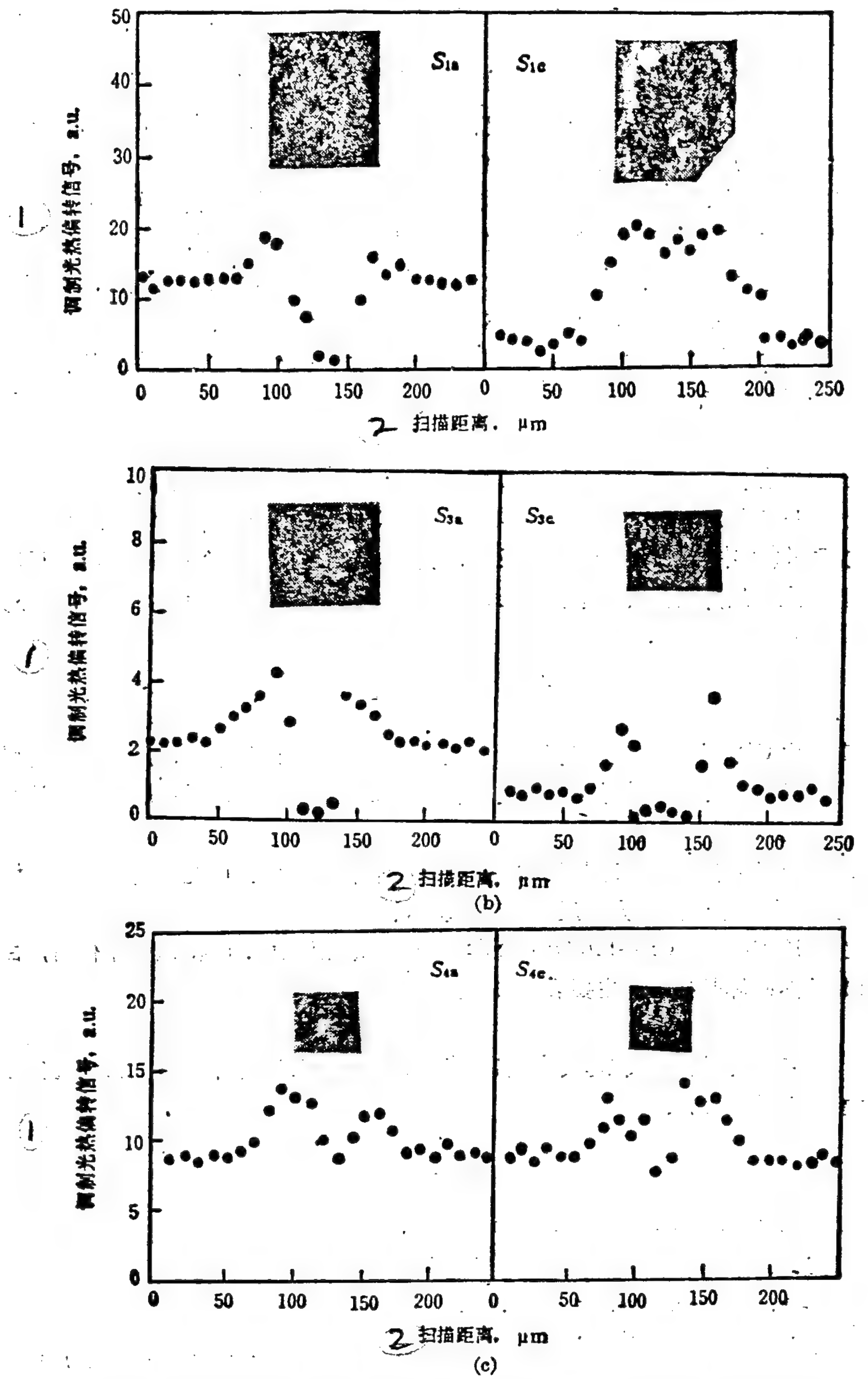


Fig.3 An illustration of the principle of detection of the damage position by pulsed time-resolved photothermal deflection technique.

Comparing Fig.2(a) and Fig.2(b), it is easy to see that, in the case of TiO_2 single layer films, substrates laser pretreated in a vacuum cause the locations of occurrence of initial damage points to move from the thin film-substrate boundary layer toward the interior of the film layer. Going a step further, the explanation is that irradiation pretreatment improves boundary surface structure. However, with regard to TiO_2/SiO_2 high reflection film, due to light fields being strongest in the outermost layers of high refractive index media, the result is that destruction always occurs in the interior of this layer. There is no relationship to whether or not substrate underwent irradiation processing. These conclusions are in line with the analysis of section 3.1 above.

In Fig.3, 1 and 2 are damage locations. 3 is the damage laser beam. 4 is the survey probe laser beam. S_0 is the optical pulse signal. S_1 is the photothermal deflection signal peak value corresponding to damage location 1. S_2 is the photothermal deflection signal peak value corresponding to damage location 2. On the basis of the references [18],

$$t_2 : t_1 = x_2^2 : x_1^2.$$



(a) TiO_2 single layer; (b) SiO_2/TiO_2 AB coating; (c) TiO_2/SiO_2 HB coating.

Fig.4 Analysis of the damage morphology

Key: (1) Modulation Photothermal Deflection Signal
(2) Scanning Distance

3.4 Sample Damage Morphology Analysis

In order to analyze a step further the influence of substrate laser pretreatment on optical thin film behavior, TiO_2 single layer film, SiO_2/TiO_2 AR coating, as well as TiO_2/SiO_2 HR coating are taken as examples. Using modulation photothermal deflection techniques as well as Nomarski optical microscopes, analyses were done of damage morphology. Results are as shown in Fig.4. The dotted line (dots) in Fig.4 are scattered sampling points.

(1) In Fig.4(a), with regard to TiO_2 single layer films, as far as samples associated with substrates which have not gone through irradiation treatment are concerned, the damage occurs on the boundary surface between film and substrate. The damage displays a morphology which has substrate and boundary surface film layers being ablated. Going a step further, this leads to the entire film layer peeling off. This causes modulation photothermal deflection signals at the center of damage points to almost fall to zero. In the case of samples associated with substrates which have gone through irradiation processing, the damage occurs in the interior of film layers. The damage displays a form which has increases in modulation photothermal deflection signals, clearly showing that damage area residual film layer absorption is markedly strengthened.

(2) In Fig.4(b), with regard to SiO_2/TiO_2 AR coatings, the two types of sample damage both occur on film and substrate boundary surfaces. This clearly shows that even though substrates go through laser irradiation pretreatment in a vacuum, AR coating film and substrate boundary surfaces are also still the weak links most easily damaged.

Carefully comparing the damage morphologies associated with the two types of samples, it is also possible to see that, with

the same type of pulse laser function, sample damage spots associated with substrates not having gone through irradiation treatment are relatively large. Moreover, substrates also have slight destruction. This is due to the boundary surface quality being relatively bad.

(3) In Fig.4(c), with regard to TiO_2/SiO_2 HR coatings, damage morphologies associated with the two different types of samples are completely the same. This clearly shows that HR coating initial damage primarily occurs in the outermost layers of high refractive index media layers. As far as sample damage behavior is concerned, influences felt in association with film layer-substrate boundary surfaces are not great.

4. CONCLUSIONS

(1) Substrates CO_2 laser pretreated in a vacuum are capable of eliminating the adsorption of such impurities as substrate surface water and so on, thus improving thin film sample film layer-substrate surface quality. This type of surface quality improvement has no great influence on HR coatings. In the case of single film layers as well as AR coatings, by contrast, it is possible to lower absorption losses and raise damage threshold values.

(2) Substrates CO_2 laser pretreated in a vacuum have relatively great influences on thin film uniformities. Because of this, this type of technology must really be made practical use of. There is a need for further steps to improve irradiation laser beam quality as well as irradiation forms.

(3) In the process of media film damage (10ns-1.06 micron-Nd:YAG laser), absorption takes the dominant role. Thermal

destruction arising from localized absorption is the main form of damage appearing.

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